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MAIL STOP AMENDMENT
Commissioner for Patents
P.O. Box 1450
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SUBMISSION OF PRIORITY DOCUMENT

Attached please find the certified copies of the following foreign applications from which priority is claimed for this case:

Country	Application Number	Filing Date
EP	99111209.5	June 9, 1999

Respectfully Submitted,

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Bescheinigung

Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

Certificate

The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Attestation

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. **Patent application No.** **Demande de brevet n°**

99111209.5 / EP99111209

The organization code and number of your priority application, to be used for filing abroad under the Paris Convention, is EP99111209

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

Le President de l'Office européen des brevets
p.o.

R.C. van Dijk

RV82451

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Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
If no title is shown please refer to the description.
Si aucun titre n'est indiqué se referer à la description.)

New esters and ester compositions

In anspruch genommene Priorität(en) / Priority(ies) claimed / Priorité(s) revendiquée(s)
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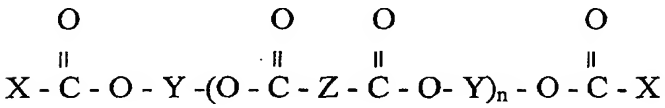
NEW ESTERS AND ESTER COMPOSITIONS

The present invention is related to new esters and ester compositions based on a polyol, a dicarboxylic acid and a monocarboxylic acid.

Such esters are known from WO99/16849, which describes esters having a kinematic viscosity at 100° C ($V_{K,100}$) of 30 cSt and higher.

Surprisingly, it was found that by using a specified diol, dicarboxylic acid and monocarboxylic acid an ester or ester composition may be obtained which has a low viscosity at 40°C and 100°C and which is suitable as lubricant in metal working fluids, in particular rolling fluids, and in hydraulic fluids. Therefore the present invention is concerned with an ester or ester composition according to the formula :



wherein X is an aliphatic hydrocarbyl group having 5 - 11 carbon atoms;

Y is an alkylene group having 2 - 8 carbon atoms;

Z is an aliphatic hydrocarbyl group having 3 - 5 carbon atoms and

n is a weight average number between 1 and 10.

X may be saturated or unsaturated and linear or branched. Preferably X is saturated and linear. Most preferably X has 7 - 9 carbon atoms. Examples of X are groups obtained after removal of the -COOH group in hexanoic acid, heptanoic acid, nonanoic acid and, in particular, octanoic and decanoic acid and mixtures thereof.

Y may be saturated or unsaturated and linear or branched. Preferably it is saturated and branched. Most preferably Y has 4 - 6 carbon atoms. Y may be

derived from e.g. ethylene glycol, propylene glycol, butane diol, pentane diol, hexane diol, heptane diol, octane diol and in particular from neopentyl glycol, and mixtures thereof.

Z may be saturated or unsaturated, linear or branched and preferably is linear and saturated. Most preferably Z has 4 carbon atoms. Z may be derived, by removal of the two -COOH groups, from glutaric acid, pimelic acid and preferably adipic acid, and mixtures thereof.

n preferably is 1.5 - 5.

The compounds and compositions, according to the present invention, may be used as lubricants in metal working fluids, more particularly in rolling fluids; they show an improved lubricating and clean burning performance. Further, they may be used as lubricants in hydraulic fluids showing amongst others a low viscosity at low temperature, a good stability regarding oxydation, a good thermal stability and biodegradability. The kinematic viscosity of the esters preferably is below 20 cSt at 100° C and below 150 cSt at 40° C.

The esters and ester compositions, according to the present invention, are prepared by reacting a monocarboxylic acid with group X, with a diol with group Y and a dicarboxylic acid with group Z. The reaction may be conducted under reduced pressure and slightly elevated temperature, but is preferably conducted at atmospheric pressure at 200 - 250° C.

The relative amounts are so chosen as to obtain the esters or ester compositions with the indicated value for n. Those skilled in the art will be able to determine these relative amounts easily, certainly in the light of the examples.

Preferably the ratio of OH groups and COOH groups in the reaction mixture, at the start of the reaction, is 0.9:1 - 1.1:1 and most preferably 0.95:1 - 1.05:1. Ideally, this ratio is about 1:1.

The ratio of COOH groups from the monocarboxylic acid and from the dicarboxylic acid in the reaction mixture, at the start of the reaction, preferably is 0.3:1 - 1.5:1 and most preferably 0.4:1 - 1:1.

During the reaction water and some monocarboxylic acid and diol are distilled; the monocarboxylic acid is being fed back into the reaction mixture and the water containing the diol is removed.

The reaction is continued till the hydroxyl value is below 15 mg KOH/g.

After the preparation, any excess of monocarboxylic acid is removed by distillation.

The ester and ester compositions according to the present invention may be used in lubricants in particular in metal working and especially in rolling fluid; as base fluids or additives in such metal working and especially rolling fluids wherein such base fluids or additives comprise other ingredients commonly used in metal working and especially in rolling fluids and known to those skilled in the art and especially those selected from mineral oil, oils of vegetable and/or animal origin and other synthetic esters, surfactants, emulsifiers, corrosion inhibitors, anti-oxidants, anti-wear/EP-agents and anti-foaming agents; the amount of the esters and ester compositions according to the present invention in such base fluids ranging from 5 to 70 and preferably from 10 to 50% by weight calculated on the weight of the base fluid. Further such esters and base fluids may be used in metal working and especially rolling fluids comprising water wherein the amount of ester or base fluid: water is 99:1 to 1:99 and preferably 20:80 to 2:98.

The ester and ester compositions according to the present invention may further be used as or in hydraulic fluids comprising up to 5% by weight and preferably 2-5% by weight of other ingredients commonly used in hydraulic fluids and known to those skilled in the art and especially those selected from the ones mentioned above.

Example 1

21 kg of a mixture of octanoic and decanoic acid ($^{60}/_{40}$ %, $^{w}/_{w}$), 20 kg of neopentyl glycol and 19 kg of adipic acid were fed into an autoclave, mixed,

heated to 230° C at atmospheric pressure and allowed to react. The process was conducted under a N_2 blanket. During the reaction water formed and some monocarboxylic acid were distilled off. The monocarboxylic acid was fed back into the reactor. The reaction was allowed to proceed till the OH-value was below 15 mg KOH/g. Then, excess of monocarboxylic acid was removed by applying vacuum (400 - 500 mbar).

Finally, the mixture was stripped under N_2 (1 hour, 230° C, 400 - 500 mbar), cooled to 80° C and, after addition of 125 g of Dicalite ™ 478 (filter aid from Lafarge Redland Minerals), filtered. 6.9 kg of water, 1.6 kg of monocarboxylic acid and 51.5 kg of an ester according to the invention was obtained. This ester showed an n of about 3.5 and had a kinematic viscosity at 40 and 100° C of 91 and 13 cSt respectively and an acid value of 28 mg KOH/g.

Example 2

Example 1 was repeated with 26.1 kg of the octanoic/decanoic acid mixture, 19 kg of neopentyl glycol and 14.9 kg of adipic acid.

The product obtained (52.6 kg and 6.6 kg of water and 0.8 kg of monocarboxylic acid) was an ester according to the invention and showed an n of about 2.2 and had a kinematic viscosity at 40 and 100° C of 46 and 8 cSt respectively and an acid value of 21 mg KOH/g.

Example 3

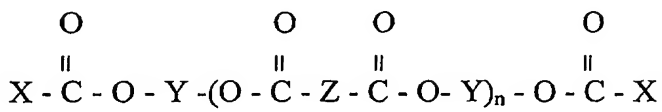
Example 1 was repeated with 18.2 kg of neopentyl glycol, 13.7 kg of adipic acid and 24.2 kg of the octanoic/decanoic acid mixture. The reaction was conducted at 225° C for about 8 hours; when the acid value was below 20 mg KOH/g (after about 5 hours) 2.7 gram of catalyst (20 parts by weight of tetrabutyl titanate in 80 parts by weight of di-2-ethylhexyl-azelate) was added.

6.1 kg of water and 50.0 kg of an ester according to the invention (with n is about 2.1) was obtained. The ester had a kinematic viscosity at 40 and 100° C of 45 and 8 cSt respectively and an acid value below 1 mg KOH/g.

5

CLAIMS

1. Ester or ester composition according to the formula :



wherein X is an aliphatic hydrocarbyl group having 5 - 11 carbon atoms;

Y is an alkylene group having 2 - 8 carbon atoms;

Z is an aliphatic hydrocarbyl group having 3 - 5 carbon atoms and

n is a weight average number between 1 and 10.

2. Ester according to claim 1 wherein the ester has a kinematic viscosity at 100° C of less than 20 cSt and a kinematic viscosity at 40° C of less than 150 cSt.

3. Ester according to claims 1 - 2
wherein
X is saturated and linear and has 7 - 9 carbon atoms,

Y is saturated and branched and has 4 - 6 carbon atoms,

Z is saturated and linear and has 4 carbon atoms, and

n is 1.5 - 5.

4. Use of an ester or ester composition according to claims 1 - 3 as or in hydraulic fluid or metal working fluid.

5. Process for preparing an ester or ester composition according to claims 1 - 3, by reacting a monocarboxylic acid with group X, a diol with group Y and a dicarboxylic acid with group Z, wherein the ratio of OH-groups and COOH-groups in the reaction mixture, at the start of the reaction, is 0.9:1 - 1.1:1 and the ratio of COOH-groups from the monocarboxylic acid and the dicarboxylic acid in the reaction mixture, at the start of the reaction, is 0.3:1 - 1.5:1.

ABSTRACT

Ester based on a monocarboxylic acid, a dicarboxylic acid and a diol and its use as or in hydraulic and metal working fluids.

5